Syllabus, Chemistry 231-01, -03, -05 Fall '09

Disclaimer: this syllabus is tentative and may be subject to change if circumstances beyond my control require it.

Contact Information

Instructor name: Dr. Thomas Junk
Time of class: 2:00-5:00 p.m. M,T,R
Office hours: see below
Instructor's e-mail: junk@ulm.edu
http://www.ulm.edu/~junk/home.html
Room of class: CNSB 244 (pre-lab) and 240
Instructor's office: CNSB 112
Office phone: 342-1830

My schedule	Mo	Tu	We	Th	Fr
8:00-8:50 a.m.	230-02		230-02		230-02
9:00-10:00 a.m.	Office hr	Office hr	Office hr	Office hr	Office hr
2:00-5:00 p.m.	231-01	231-03		231-05	
5:00-6:00 p.m.		230 Review*			

* No reviews held during the first week of class. Location for review sessions TBA. Timing flexible to accommodate exams and teaching schedules. Participation not mandatory.

Course Prerequisites/Corequisites

You must have passed, or currently attend, Chem 230. If you drop Chem 230 you need **special permission** to stay in Chem 231!

Course Description, Objectives and Outcomes

You will complete 10 lab sessions during which you will perform experiments. In addition, you will attend check-in (today) and check-out (after the final). See attachment of details.

Course Topics

See the attached schedule for all topics covered by the experiments.

Instructional Methods and Activities

This course familiarizes students with basic laboratory skills in organic synthesis and supplements Chem 230 by introducing applied techniques. Specifics of each experiement to be conducted are attached. You will work **on your own**.

Dress code: **No exposed skin** from the chest down, no ultra-thin fabrics. You must wear closed, sturdy shoes that protect your feet **and heels** adequately (tennis shoes are a good choice). You must wear eye protection **whenever** you are in the lab. Make sure your clothing and hairstyle is compatible with a lab environment. If you violate the dress code, you will be sent home to change and will very likely miss the respective lab. **You should keep spare compliant shoes, pants, and a shirt in your drawer** in case of an accident.

Evaluation and Grade Assignment

Tests, quizzes: Each quiz will cover mostly material from the **previous** pre-lab, as well as the book chapter corresponding to that day's experiment. Also, expect one simple, applied question relating to **today's** experiment. Bring a calculator for your quizzes and final! Quiz dates are attached.

Readings: You are expected to read the attached procedure and any assigned book chapters for the day's experiment **in advance.** Be prepared to answer questions related to these procedures. If you have any questions as to which material you will have to review it is your responsibility to ask.

Homework assignments: Lab reports are due one week after each experiment and have to be completed at home if necessary. I expect all lab reports to be entered into a carbon copy lab notebook.

Grading system: Your grade will be based on 520 points total:

10 quizzes (15 pts each, 2 can be dropped except safety)	120 points
10 experiments (lab reports worth 25 pts each)	250 points

1 final 150 points

Grades will be assigned based on the usual scale (A, 100-90%; B, 89-80% etc.). There will be no curving and you cannot drop any reports.

Mid-term grades: ULM requires that I post mid-term grades for you by Oct 16. **Mid-term grades indicate a student's status at mid-semester only and do not indicate the final performance outcome for a student!**

Class Policies and Procedures

Textbooks and materials: Williamson's *Microscale and Macroscale Organic Experiments* and any material covered by the hand-outs. Other required material: Model Kit, calculator, eye protection, proper dress code, carbon copy lab notebook.

Attendance policy: You must be present for the entire duration of all pre-labs and experiments. Any unexcused absences will result in 0 points earned for the missed report and quiz.

Make-up policy: You may miss one experiment as a consequence of an excused absence, but generally must make this up on the assigned day (see attachment). Any misses require a written excuse (e.g. from your physician).

Academic integrity: If you cheat, you will get zero points on the exam or quiz. This may happen to you even if you are a cooperating "benefactor": it is in your interest not to cooperate. If you are caught cheating on the final you will be 150 points short and usually will not be able to pass.

Course evaluation policy: Please complete the on-line course evaluation!

Student Services: Please note that the following ULM student services are available to you: Student Success Center (*http://www.ulm.edu/cass/*), Counseling Center (*http://www.ulm.edu/counselingcenter/*), Special Needs (*http://www.ulm.edu/counselingcenter/special.htm*).

Discipline, course specific policies: If your cell phone rings you may face a penalty (typically, a point loss). Only **simple** (non-programmable) calculators are allowed or needed during exams. **All experiments have to be carrid out by you only (no partners!)**

Office hours and review sessions: Office hours are intended to discuss problems with your schedule, allow you to see your quizzes, etc. You may attend my 231 review sessions if you have any problems with the theory. In practice, there usually is plenty of time to discuss your questions in lab.

Posting of exam results and answer keys: Quiz results will be posted next to my door, according to your student ID number. Copies of quizzes will be posted at *http://www.ulm.edu/~junk/coursestaught.html*.

Cancellations: Cancellations are rare. Cancelled labs should be treated like labs that were never scheduled: If your lab is cancelled, your next quiz will cover the last pre-lab before the cancellation, your lab reports will be due the following lab, your grade will be based on the number of labs offered to you, and your next experiment will take place as scheduled.

Come prepared when we meet again! By next time, you should a) follow the dress code, b) have your goggles or safety glasses, c) be prepared for your first quiz, d) bring your carbon copy lab note book, e) bring your lab text book (Williamson's), f) bring or remember your combination.

Lab Schedule
Organic Chemistry 231

#	M	T	R	Chapter	Title
0	8/24	8/25	8/27	**1,2,& glassware**	Introduction, Safety, Grading, Check-In
1	8/31	9/1	9/3	**3**	Melting Point
2	9/14	9/8	9/10	**4**	Recrystallization
3	9/21	9/15	9/17	**41**	Aspirin-Reactions Calculation
4	9/28	9/22	9/24	**5**	Distillation
5	10/5	9/29	11/1	**5**	Fractional Distillation
6	10/12	10/6	10/8	**7**	Extraction
7	10/19	10/13	10/15	**58**	Alkene Addition
8	11/2	10/27	10/29	**19**	Cyclohexene (E1)
9	11/9	11/3	11/5	**17**	2-Chloro-2-methylpropane (S_N1)
10	11/16	11/10	11/12	**17**	Phenoxyacetic Acid (S_N2)
11	11/23	11/17	11/19	**All**	Check-out/make-up
Final	11/30	12/1	12/3	**All**	Final Exam

CHEMISTRY 231--Desk and Kit contents

Student Name_____

Student Number_____

Section # _____ Room # _____ Desk # _____ Combination ___-___-___

Item	#	Item	#
Beaker, 50 mL	2	Keck clamps*	4
Beaker, 100 mL	2	Graduated cylinder, 10 mL	1
Beaker, 400 mL	1	Graduated cylinder, 100 mL	1
Erlenmeyer flask, 250 mL	1	Spatula	1
Erlenmeyer flask, 125 mL	2	Stirring bar, magnetic*	1
Erlenmeyer flask, 50 mL	3	Stirring rod, 6 inch	1
Filter flask, 125 ml	1	Test tubes, 15 x 125 mm	4
Buchner funnel with stopper	1	Thermometer	1
Long-stem funnel	1	Watch glass	1
Powder funnel			

Organic Kit

Quantity	Description
1	Adapter, Claisen*
1	Adapter, rubber*
1	Adapter, thermometer, w/rubber adapter*
1	Adapter, three-way*
1	Adapter, vacuum, 105 º*
1	Condenser, West, 190 mm*
1	Distilling column, 190 mm*
1	Flask, single neck, 25 ml*
1	Flask, single neck, 50 ml*
1	Flask, single neck, 100 ml*
1	*Flask, three neck, 500 ml* (not needed, may be absent)*
1	Funnel, separatory, 125 ml, w/Teflon® stopcock*
2	Stopper, glass*
1	Foam insert
1	Case

*Replacements kept in Room 240A (all other replacements kept in Room 122).

SAFETY AGREEMENT

I, _____(Print Name), have read, understand, and agree to the laboratory Safety Rules. I, therefore, release the Department of Chemistry at ULM and my instructor from any responsibility for accidents occurring while not following those rules.

_____Signed _____Dated

EXPERIMENT 1: MELTING POINTS

1. Prepare a known sample of urea and determine its melting point. (Use a well packed capillary with ~1-2 mm of sample, with a rise in temperature of ~ 1-2 ^{o}C per min near the mp.) Always record the temperature as a RANGE; the temperature at which the sample first melts until it is completely melted.

2. Prepare an unknown sample, determine its melting point, and identify it.

3. Perform a mixed-melting point experiment by preparing a 50%-50% mixture of unknown with the suspected known and determine if a melting point depression occurs. If it does, your identification was incorrect and you have to have to repeat the assignment.

EXPERIMENT 2: RECRYSTALLIZATION

1. Weigh about 2.0 grams of benzil (record accurately to 0.000 g) and transfer the benzil to a 125 mL Erlenmeyer flask and add 20 mL of methanol.

2. Using a hot plate-stirrer and a magnetic stirring bar, heat the flask until all the benzil dissolves.

3. Remove the flask from the heat and place on the lab bench. Allow the flask to return to room temperature (RT) **WITHOUT DISTURBING** the flask.

4. After cooling to RT, cool the flask in an ice bath and collect the crystals by vacuum filtration using a clamped filter flask attached to an aspirator and a Buchner funnel with a piece of filter paper.

5. Remove the stem of the funnel, place the top part in your drawer and allow the crystals to dry until the next lab period and determine the melting point and the percent recovery.

EXPERIMENT 3: ASPIRIN

1. Bring 60-90 mL of water in a 400-mL beaker to boiling using a hot plate-stirrer

2. Combine 3.5 grams of salicylic acid, 3.5 mL of acetic anhydride, and 4 to 5 drops of concentrated sulfuric acid and a magnetic stir bar in a 125-mL Erlenmeyer flask. **Make sure the flask is dry!**

3. Using a 3-prong clamp, place the flask in the boiling water bath with stirring for 5 to 6 minutes.

4. Remove the flask from the boiling water bath and add approximately 15 mL of ice water.

5. Cool the flask in an ice bath and collect the solid by vacuum filtration.

6. Transfer the crystals to a 125-mL Erlenmeyer and recrystallize using 8 mL of ethanol and 20 mL of water. Collect the crystals by vacuum filtration. Allow the crystals to dry until the next lab period when the melting point and percent yield can be determined.

EXPERIMENT 4: SIMPLE DISTILLATION

1. Assemble a simple distillation apparatus according to the directions given. Use a <u>minimal</u> amount of vacuum grease on each ground glass joint. Add approximately 30 mL of the unknown and a magnetic stir bar to the 50 mL round-bottom flask.

2. Using a heating mantle, heat to boiling and adjust the heat such that the condensation line is above the entrance to the condenser but below the thermometer adapter. **Make sure you do NOT heat your stir plate! This will cause $300 in damage!**

3. Record the temperature vs. mL of distillate every 2 mL. After 25 mL of distillate has been collected, lower the heating mantle and let cool. Determine the boiling point and the identity of the unknown.

4. Return the distillate and the remaining unknown in the round-bottom flask to the waste solvent bottle in the hood.

Unknown possibilities:

Acetone	57 ºC
Hexane	69 ºC
Ethyl Acetate	78 ºC
Cyclohexane	83 ºC
Heptane	98 ºC

EXPERIMENT 5: FRACTIONAL DISTILLATION

1. Assemble a fractional distillation apparatus using 25 grams of glass beads and a small plug of copper wire as a fractionating column and add 30 mL of a 1:1 mixture of 2 unknowns.

2. Heat to boiling and adjust the heat such that the condensation line is above the entrance to the condenser but below the thermometer adapter.

3. Record the temperature vs. mL of distillate every 2 mL. After 25 mL of distillate has been collected, lower the heating mantle and let cool. Determine the boiling points and the identities of the unknowns.

4. Return the distillate and the remaining unknown in the round-bottom flask to the waste solvent bottle in the hood.

Unknown possibilities: same as before

EXPERIMENT 6: EXTRACTION

1. Familiarize yourself with the separatory funnel by filling it with water and draining the water out under the sink.

2. In a separatory funnel with the stopcock **CLOSED**, add 30 mL of t-butyl methyl ether (MTBE) (ρ = 0.740 g/mL) and approximately 3 grams of the 1:1 benzoic acid/biphenyl mixture.

3. Add 16 mL of 1 M NaOH, stopper, and shake for about 2 minutes, venting the separatory funnel frequently.

4. Using a ring clamp for the separatory funnel, allow the layers to separate.

5. Remove the aqueous (bottom) layer, placing it in a 125 mL Erlenmeyer flask.

6. Wash the organic layer with 10 mL of water, allow the layers to separate, and add the aqueous layer to the Erlenmeyer flask.

7. Transfer the organic layer to a 50- or 100- mL beaker, label the beaker with your name, and place the beaker in the hood to evaporate the MTBE.

8. Acidify the Erlenmeyer flask with concentrated hydrochloric acid by drop-wise addition until no further precipitate is formed.

9. Cool the solution in an ice bath and collect the crystals by vacuum filtration. Transfer the filter paper with the benzoic acid crystals to a watch glass and allow the crystals to dry until the next lab period and determine the melting point and the percent recovery.

10. Place the beaker containing the MTBE **under the hood** on a piece of paper stating your name until the next lab period, then scrape the biphenyl from the beaker and determine the percent recovery.

EXPERIMENT 7: MESO-1,2-DIBROMO-1,2-DIPHENYLETHANE

1. In a 50-mL Erlenmeyer flask, combine 1 gram of E-stilbene and 20 mL of acetic acid and a magnetic stir bar. Heat gently to approx. 50° over a hot plate-stirrer with stirring to dissolve the stilbene. DO NOT BOIL!!

2. Once dissolved, remove from the heat and **SLOWLY** add 2 grams of pyridinium perbromide to the warm solution and mix well. If necessary, wash the reagent down the side of the flask with additional acetic acid using a disposable pipette. Continue heating for 2 minutes with stirring after the addition, but DO NOT HEAT ABOVE 50°!!

3. Cool in an ice bath.

4. Collect the crystals by vacuum filtration. Allow the crystals to dry until the next lab period and determine the melting point and the percent yield.

EXPERIMENT 8: CYCLOHEXENE (E1)

1. Combine 20.0 mL of cyclohexanol, 5.0 mL of phosphoric acid, and a magnetic stir bar in a 50-mL round bottom flask. (Note: cyclohexanol can freeze if the lab is cold. If it does, just warm the flask with hot water)

2. Assemble a simple distillation apparatus and distill the cyclohexene with stirring. Submerge the receiving flask in an ice bath.

3. Transfer the distillate to a separatory funnel secured with a ring clamp, allow the layers to separate, and remove the aqueous layer.

4. Wash with 15 mL of a saturated sodium chloride solution, allow the layers to separate, and remove the aqueous layer.

5. Transfer the organic layer to an Erlenmeyer and add ~5 g of sodium sulfate. Stopper well and let stand for ~ 5 min.

6. Carefully decant the liquid into a round bottom flask closed with a **well-greased** stopper and determine the percent yield. The remaining solid is waste – spent drying agent!

EXPERIMENT 9: 2-CHLORO-2-METHYLPROPANE (tert-butyl chloride) (S_N1)

1. Place 0.25 mol of 2-methyl-2-propanol (tert-butyl alcohol) and 0.75 mol of HCl (from concentrated hydrochloric acid) in a 125 mL separatory funnel. (**Calculate the quantities you need first, then check your results with your colleagues and finally with your instructor so you don't have to repeat the experiment!**)

2. Shake the mixture intermittently for 20 minutes, allowing the separatory funnel to rest unstoppered in between.

3. Separate the layers and wash the organic compound independently with water (25 mL), saturated sodium bicarbonate (25 mL), water (20 mL), and saturated sodium chloride (20 mL). **Keep all phases so you don't lose your product by mistake!**

4. Transfer the organic layer to an Erlenmeyer and add ~ 2 grams of anhydrous calcium chloride. (Stopper well and let stand for ~ 5 min.)

5. Carefully decant the liquid into a round bottom flask closed with a **well-greased** stopper and determine the percent yield. The remaining solid is waste – spent drying agent!

EXPERIMENT 10: PHENOXYACETIC ACID (S_N2)

1. Place 20 mL of 2M NaOH in a 50-mL round-bottomed flask with stirring.

2. Cool the flask in an ice bath and carefully add with stirring 2.1 grams of phenol (90%), 1.9 grams of chloroacetic acid, and 0.5 grams of potassium iodide. **Both phenol and chloroacetic acid can easily burn your skin!**

3. Remove the flask from the ice bath and reflux the mixture for 1 hour.

4. Cool the reaction mixture in an ice bath, then transfer to a 125 mL Erlenmeyer flask.

5. Slowly, add 5 mL of concentrated hydrochloric acid to the cold reaction mixture.
   Product typically separates as an oil. DO NOT FILTER until product is SOLID.
   Seeding product may be required for crystallization.

6. Collect the product by vacuum filtration, washing with cold water in the Buchner funnel.